

The comparison of computer methods for porosity evaluation in sintered constructional steels

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ABSTRACT

Purpose: Purpose of this paper is to compare the results obtained from the parameters characterizing the porosity using two computer programs.

Design/methodology/approach: The powders used in the present paper are pre-alloyed iron-base powders. The amount of graphite which is mixed with the iron-base powder is 0.6% and lubricant is 0.75%. Green compacts were sintered in a vacuum furnace and cooled in nitrogen at three different rates: 6.5°C/s, 1.6°C/s and 0.3°C/s. Next the samples were tempered in vacuum in the same furnace and cooled in nitrogen, with the exception of low cooling. Obtained samples were examined by light optical microscopy (LOM) for microstructure observation and scanning electron microscopy (SEM).

Findings: Investigation of porosity in the pre-alloyed steels was performed using "Image-Pro Plus" computer program of Media Cybernetics company and "Sigma Scan" computer program of Systat Software company. Leica light microscope (MEFA4) was used to prepare series of micrographs. The microstructures were observed at 25x magnification and recorded as *.jpg files. After calibration of the digital images, the chosen parameters were calculated.

Practical implications: Both programs have some limitations, which made it difficult to obtain some results automatically.

Originality/value: The obtained results from the programs confirm that these programs are very useful in the statistical analysis of microstructures of sintered materials, because of the time savings during calculations.

Keywords: Powder metallurgy; Sintering constructional steels; Computer methods of porosity analysis

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1. Introduction

Generally known techniques applied for investigation of the sintered materials are computer-aided methods, which allow

metallographic analysis, where images were obtained from the scanning and light microscope. Next, the micrographics obtained by microscopes may be subject to digital processing using the advanced graphical programs such as: Image Pro Plus, SigmaScan, Image Tool, Aphelion, Olympus. Albeit the computer

processing of the pores data is made in 2D, yet, in the material they are the three-dimensional objects. Based on metallographic images, it is possible to investigate the pores size, their morphology, distribution of their diameter and spacing, but they are only two-dimensional images. Bigger complicated shapes and pores layout can lead to omission of the meaningful information. Irregularity of pores causes require determination a pore diameter. The term "diameter" determines only pores of nearly spherical shape. With very complicated shapes the "substitute diameter" term is applied, which determines a diameter of rough virtual object of about the same surface, as the analyzed pore [1-15].

The images from microscopes are saved in the digital form in a one of the basic methods of graphic recording: vector and raster one. The vector graphics is saved using mathematical functions and consist of points (nodes) connected with lines. Defined by nodes' coordinates and radii of curvature interconnecting elements form the particular objects. Each object is an independent part of the picture and can be modified using the appropriate programs. Raster graphics is stored in a table of numbers corresponding to the color of the particular points (pixels) that create the image. The bitmap for computer analysis is used, because it allows the identification of many more details. Each program has its own proprietary graphical format which cannot be read by most programs, products of other companies. Raster graphics is based on several formats with the most common being: tiff, bmp, jpg, gif [5,7].

Advantages of using digital image processing methods [5]:

- less chance of error,
- increase the objectivity of the obtained results,
- almost 100% reproducibility of the method used,
- lower cost in the case of repetitive routine investigations.

Disadvantages of digital processing [5]:

- high cost of implementing a single investigation,
- poor ability to assess the qualitative study of the microstructure,
- high quality images are required.

2. Experimental procedure

The aim of this paper was to perform an analysis with using two computer programs (Image-Pro Plus, SigmaScan Pro) which help to determine porosity occurring in sintered steels. Moreover, these programs are very useful in the statistical analysis of microstructures of sintered materials. Fig. 1 presents the process of obtaining investigated samples.

In the investigations two iron powders were used (Fig. 2) and lamellar graphite powder grade GR12. Next, four mixtures were prepared using laboratory mixers. Iron base powder was mixed with 0.6% of graphite. In the quantity of 0.75% the lubricant "Acrawax" was used. From prepared powder mixtures the set of samples was prepared using the uniaxial pressing die (die with free motion): rectangular (dimensions - 5 x 10 x 55 mm) for cross-section observations. The samples were compacted using 200 kN hydraulic press with two pressures: 500 and 600 MPa. Table 1 presents the setting-up samples prepared for investigations. Before the sintering process, the debinding operation was performed in nitrogen only atmosphere in the separate plain furnace. Prepared green compacts were then

sintered at 1120°C for 30 minutes in a vacuum furnace with argon backfilling and cooled in nitrogen at three different rates: high cooling (with a rate 6.5°C/s), medium cooling (with a rate 1.6°C/s) and slow cooling (with a rate 0.3°C/s with no gas added). Sintered compacts were tempered in vacuum in the same furnace and cooled in nitrogen.

Afterwards, the samples were ground with abrasive papers of grain 120-2000 $\mu\text{m}/\text{mm}^2$ and polished with diamond paste. Etched microsections were examined on the metallographic microscope Leica MEF4A with a total magnification of 25x.

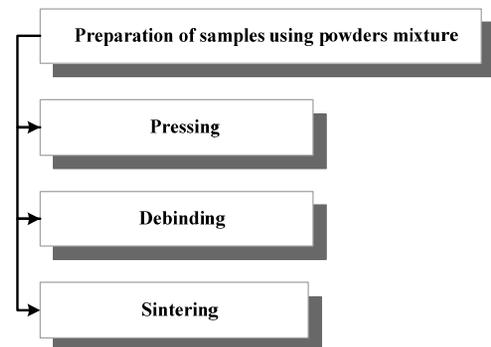


Fig. 1. Technological process of manufacturing sintered materials

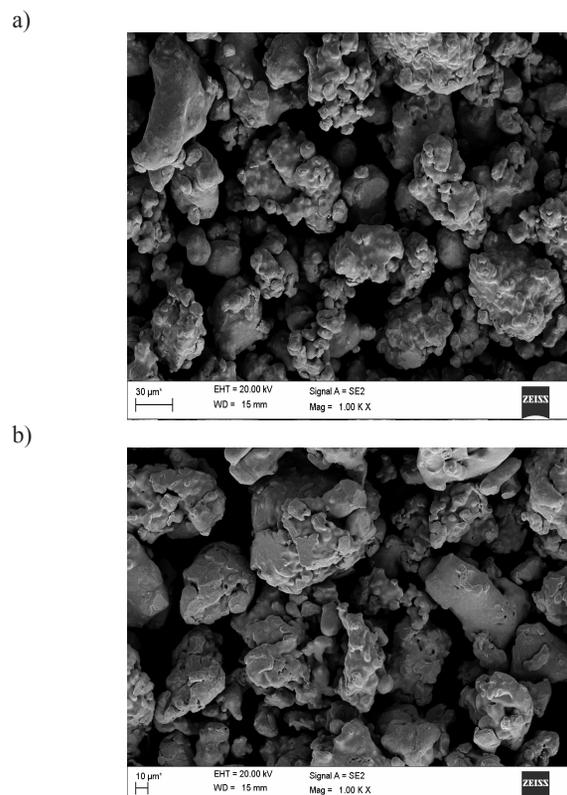


Fig. 2. Micrographs of base powders: a) Astaloy CrL pre-alloyed powder. b) Astaloy CrM pre-alloyed powder (SEM)

Table 1. The setting-up samples prepared for investigations

No	Sample symbol	Compacting pressure [MPa]	Powder type	Cooling rate [°C/s]
1	X1	500	CRL	0.3
2	X2	600		
3	X3	600		
4	X4	500	CRM	1.6
5	Y1	500		
6	Y2	600	CRL	6.5
7	Y3	600		
8	Y4	500	CRM	6.5
9	Z1	500		
10	Z2	600	CRL	6.5
11	Z3	600		
12	Z4	500	CRM	6.5

Software used

The obtained pictures were subjected to computer analysis from the point of view of porosity investigations. Image Pro Plus, Sigma Scan Pro, Image Tool belong to the most often applied programs for statistical investigations in the industry. These programs are used for computer analysis of the investigated images using the built in tools, used for analysis of the investigated images. One of available programs for digital working of images is Image-Pro Plus program, which works in Windows environment. This program accepts many graphical formats, including the four basic ones: jpg, tiff, gif, bmp. Thanks to these tools there exists a possibility for performing the standard measures: length, width, distance, medium diameter and pores spacing in the analysed sample. SigmaScan Pro program is one of many programs offered by Systat Software company. This program allows measurements such as: distances, angles, areas, classification of investigated elements, as well contains a number of tools to correct the quality of the investigated image. The layer mechanism allows both measurement areas of the specified colour, as well as areas of colour of higher intensity that the assumed threshold. The program includes a few tools for identification of edges, modification of colour palette, saturation, brightness, as well as conversion to grey scale. In the program, except of the program analyser, the is built in a useful calculation sheet, in which the obtained information are saved as well a chart diagram. Built in calculation sheet lets to perform the automatic statistical investigations of information saved in a column, e.g.: arithmetic mean, standard deviation, max. and min. values of the selected parameters. Program processes many basic graphic files, like: *.tif, *.bmp, *.jpg, *.gif.

3. Results and discussion

Based on images of powders Astaloy CrL and CRM acquired from the electron scanning microscope the globular shape of the

powder particles were observed. The results obtained for the selected parameters of pores were statistically analysed and presented in tables and plots.

Pore surface

For X - series of samples cooled with the cooling rate of 0.3 °C/s pores of the largest area were obtained for the a X1 sample, whose values were 97.16 and 76.55 μm², respectively for the ImagePro Plus software, and SigmaScan. The smallest pores are presented in the X3 sample - 42.32 μm², and X4 -19.75 μm², respectively, for ImagePro Plus and SigmaScan program. The average values of surface pore for a series of X are 64.85 μm² and 50.20 μm², respectively, for ImagePro Plus and SigmaScan software (Table 2). Fig. 3 presents an example of assessing the relationship of pores' area with different cooling rates, based on two programs.

Table 2. Assessment of the selected statistical values of the calculated X pore surface series of samples obtained using ImagePro Plus and SigmaScan software (example)

	ImagePro Plus program		SigmaScan program	
	Sample symbol	Value	Sample symbol	Value
Medium surface of pores	max. [μm ²]	X1 97.16	X1	76.55
	min. [μm ²]	X3 42.32	X4	31.16
Medium value of series X [μm ²]		64.85		50.20

For Y - series samples cooled with the cooling rate of 1.6 °C/s pores of the largest area were obtained for the Y4 sample, whose values are 41.62 and 31.02 μm², respectively, for the ImagePro Plus software, and SigmaScan. The smallest pores occur in the Y1 sample - 30.37 μm², and Y3 - 21.61 μm², respectively for ImagePro Plus and SigmaScan program. The average values of surface pore for a series of Y are 35.9 μm² and 26.21 μm², respectively, for ImagePro Plus and SigmaScan software (Table 3).

Table 3. Assessment of selected statistical values calculated for Y pore surface series samples obtained using ImagePro Plus and SigmaScan software (an example)

	ImagePro Plus program		SigmaScan program	
	Sample symbol	Value	Sample symbol	Value
Medium surface of pores	max. [μm ²]	Y4 41.62	Y2	31.02
	min. [μm ²]	Y1 30.37	Y3	21.61
Medium value of series Y [μm ²]		35.9		26.21

For Z - series samples cooled with the cooling rate of 6.5 °C/s pores of the largest area were obtained for the Z1 sample, whose

values are 60.18 and 51.97 μm^2 , respectively, for the ImagePro Plus software, and SigmaScan. The smallest pores occur in the Z3 sample - 26.36 μm^2 , and Z3 - 19.45 μm^2 , respectively for ImagePro Plus and SigmaScan program. The average value of surface pore for a series of Z are 46.22 μm^2 and 38.55 μm^2 , respectively, for ImagePro Plus and SigmaScan software (Table 4).

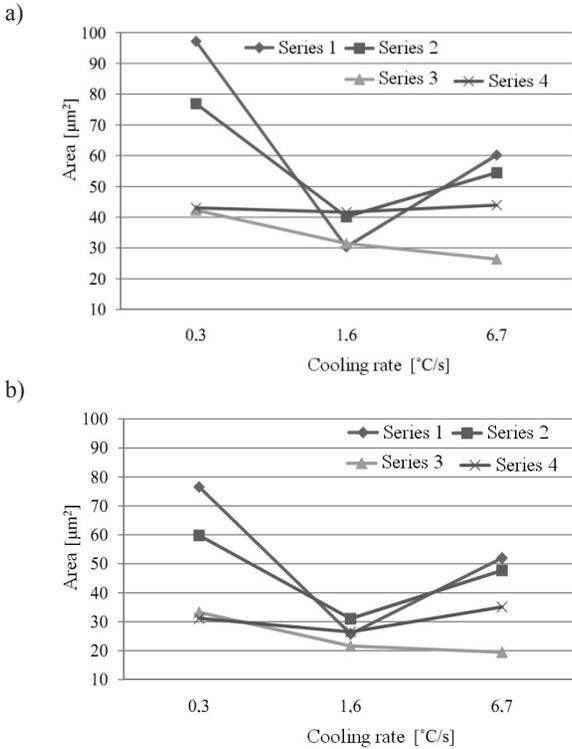


Fig. 3. Plot of the dependence values of pores' area at different cooling rate based on: a) ImagePro Plus and b) SigmaScan program (where series 1 - X1,Y1,Z1; series 2 - X2,Y2, Z2; series 3- X3,Y3,Z3; series 4 - X4,Y4,Z4)

Table 4. Assessment of selected statistical values calculated for Z pore surface series samples obtained using ImagePro Plus and SigmaScan software (an example)

		ImagePro Plus program		SigmaScan program	
		Sample symbol	Value	Sample symbol	Value
Medium surface of pores	max. [μm^2]	Z1	60.18	Z1	51.97
	min. [μm^2]	Z3	26.36	Z3	19.45
Medium value of series Z [μm^2]			46.22		26.21

Diameter of the pore

For X - series samples cooled with the cooling rate of 0.3 °C/s pores of the largest diameter were obtained for the X1 sample,

whose values are 7.29 and 7.95 μm , respectively, for the ImagePro Plus software, and SigmaScan. The smallest pores diameter occur in the X4 sample - 5.70 μm , and X4 - 6.14 μm , respectively for ImagePro Plus and SigmaScan program. The average values of surface pore for series of X are 6.53 μm and 7.10 μm , respectively, for ImagePro Plus and SigmaScan software (Table 5). Fig. 4 presents an example of assessment the dependence of pores' diameter at different cooling rates obtained with the two programs.

Table 5. Assessment of selected statistical values calculated for X pore diameter series samples obtained using ImagePro Plus and SigmaScan software (example)

		ImagePro Plus program		SigmaScan program	
		Sample symbol	Value	Sample symbol	Value
Medium diameter of pores	max. [μm]	X1	7.29	X1	7.95
	min. [μm]	X4	5.70	X4	6.14
Medium value of series X [μm]			6.53		7.10

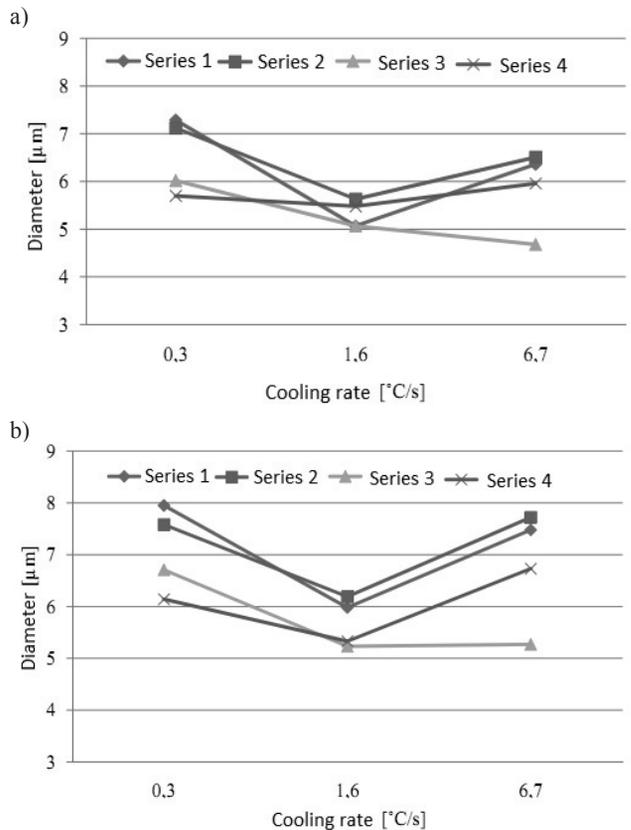


Fig. 4. Graph of the dependence values of pores' diameter at different cooling rate based on: a) ImagePro Plus and b) SigmaScan program (where series 1 - X1,Y1,Z1; series 2 - X2,Y2, Z2; series 3 - X3,Y3,Z3; series 4 - X4,Y4,Z4)

For Y - series samples cooled with the cooling rate of 1.6 °C/s pores of the largest diameter were obtained for the Y2 sample, whose values are 5.63 and 6.19 μm, respectively, for the ImagePro Plus software, and SigmaScan. The smallest pores diameter are presented in the Y3 sample - 5.07 μm, and Y3 - 5.29 μm, respectively, for ImagePro Plus and SigmaScan program. The average values of surface pore for series of Y are 5.31 μm and 5.70 μm, respectively, for ImagePro Plus and SigmaScan software (Table 6).

Table 6. Assessment of selected statistical values calculated for Y pore diameter series samples obtained using ImagePro Plus and SigmaScan software (example)

		ImagePro Plus program		SigmaScan program	
		Sample symbol	Value	Sample symbol	Value
Medium diameter of pores	max. [μm]	Y2	7.63	Y2	6.19
	min. [μm]	Y3	5.07	Y3	5.29
Medium value of series Y [μm]		5.31		5.70	

For Z - series samples cooled with the cooling rate of 6.5 °C/s pores of the largest diameter were obtained for the Z2 sample, whose values are 6.51 and 7.72 μm, respectively, for the ImagePro Plus software, and SigmaScan. The smallest pores diameter are presented in the Z3 sample - 4.68 μm, and Z3 - 5.27 μm, respectively for ImagePro Plus and SigmaScan program. The average values of surface pore for a series of Z are 5.58 μm and 6.80 μm, respectively, for ImagePro Plus and SigmaScan software (Table 7).

Table 7. Assessment of selected statistical values calculated Z pore diameter series samples obtained using ImagePro Plus and SigmaScan software (example)

		ImagePro Plus program		SigmaScan program	
		Sample symbol	Value	Sample symbol	Value
Medium diameter of pores	max. [μm]	Z2	6.51	Z2	7.72
	min. [μm]	Z3	4.68	Z3	5.27
Medium value of series Z [μm]		5.58		6.80	

Pore perimeter

For X - series samples cooled with the cooling rate of 0.3 °C/s pores of the largest perimeter were obtained for the X1 sample, whose values are 33.18 and 44.77 μm, respectively, for the ImagePro Plus software, and SigmaScan. The smallest pores perimeter are presented in the X4 sample - 22.11 μm, and X4 - 27.11 μm, respectively, for ImagePro Plus and SigmaScan program. The average values of surface pore for a series of X are 27.54 μm and 35.92 μm,

respectively, for ImagePro Plus and SigmaScan software (Table 8). Fig. 5 presents an example setting up the dependence of pores' perimeter at different cooling rates based on two programs.

For Y - series samples cooled with the cooling rate of 1.6 °C/s pores of the largest perimeters were obtained for the X1 sample, whose values are 21.70 and 27.44 μm, respectively, for the ImagePro Plus software, and SigmaScan. The smallest pores perimeters are presented in the Y1 sample - 19.00 μm, and X3 - 23.30 μm, respectively, for ImagePro Plus and SigmaScan program. The average values of pored surface for series of Y are 20.53 μm and 25.35 μm, respectively, for ImagePro Plus and SigmaScan software.

Table 8. Assessment of selected statistical values calculated X pore perimeter series samples obtained using ImagePro Plus and SigmaScan software (example)

		ImagePro Plus program		SigmaScan program	
		Sample symbol	Value	Sample symbol	Value
Medium perimeter of pores	max. [μm]	X1	33.18	X1	44.77
	min. [μm]	X4	22.11	X4	27.86
Medium value of series X [μm]		27.54		35.92	

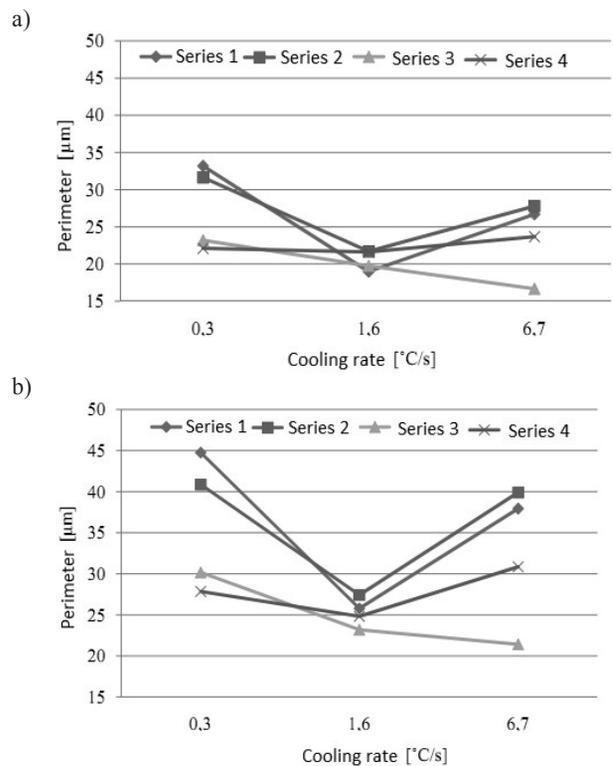


Fig. 5. Graph of the dependence values of pores' perimeter at different cooling rate based on: a) ImagePro Plus and b) SigmaScan program (where series 1 - X1,Y1,Z1; series 2 - X2,Y2, Z2; series 3- X3,Y3,Z3; series 4 - X4,Y4,Z4)

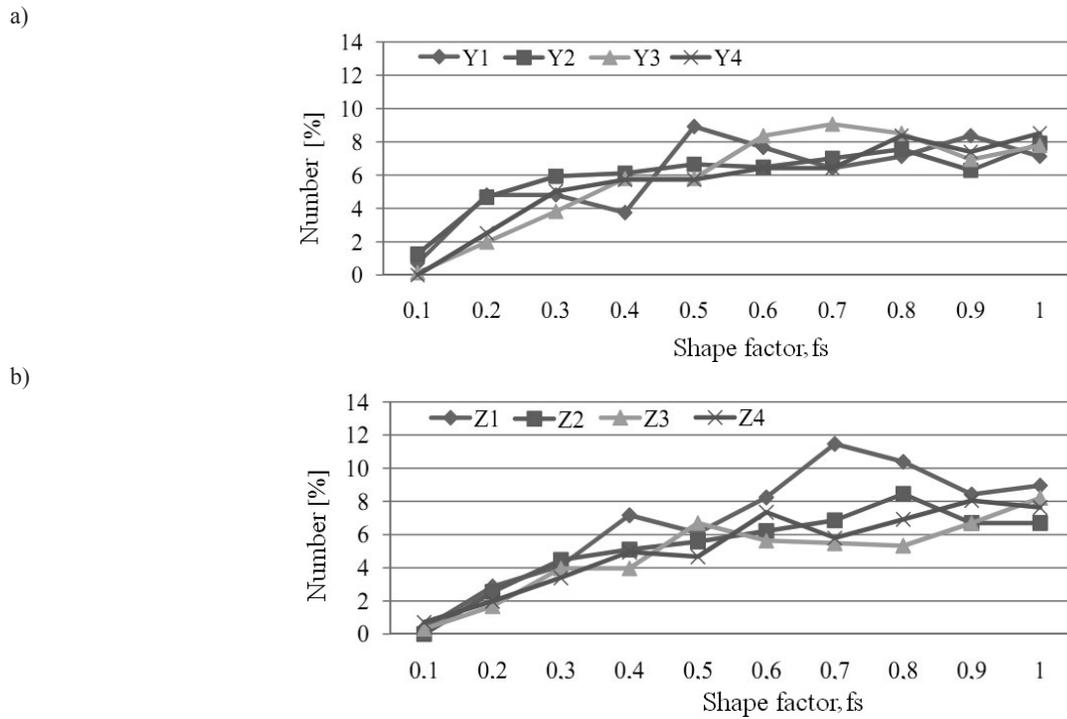


Fig. 6. Effect of four cooling rates a) medium cooling (1.6°C/s), b) rapid cooling (6.5°C/s), on distribution of the dimensionless shape factor - fs using ImagePro Plus program

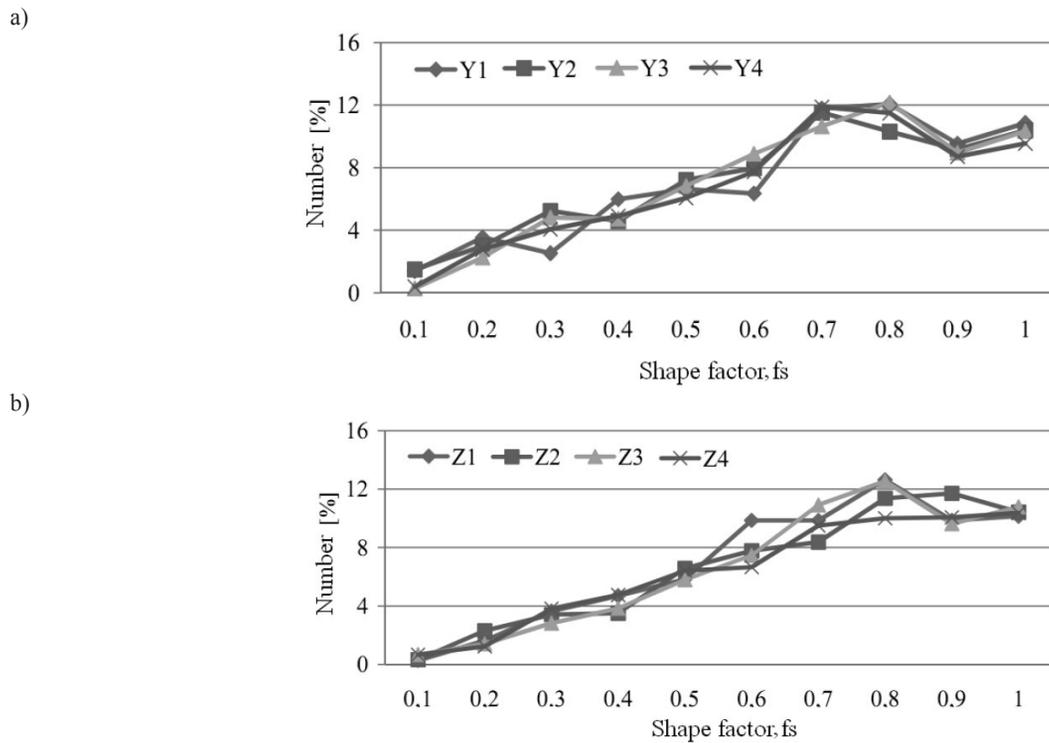


Fig. 7. Effect of four cooling rates a) medium cooling (1.6°C/s), b) rapid cooling (6.5°C/s), on distribution of the dimensionless shape factor - fs using SigmaScan program

For Z - series samples cooled with the cooling rate of 6.5 °C/s pores of the largest perimeter were obtained for the Z2 sample, whose values are 27.80 and 39.31 μm, respectively, for the ImagePro Plus software, and SigmaScan. The smallest pores perimeters are presented in the same Z3 sample - 16.67 μm, and 21.42 μm, respectively, for ImagePro Plus and SigmaScan program. The average values of surface pore for a series of Z are 23.71 μm and 32.38 μm, respectively, for ImagePro Plus and SigmaScan software.

Tables 9 and 10 present an exemplary assessment of the statistical values for perimeter tested pores obtained using two programs.

Table 9. Assessment of statistical values for perimeter tested pores obtained using from ImagePro Plus program (example)

Sample symbol	Arithmetic mean, μm	Value, μm		Standard deviation, μm	Confidence interval, μm
		Min.	Max.		
X1	33.18	26.43	42.81	7.63	26.49-39.87
X2	31.65	26.80	37.08	3.68	28.42-34.88
X3	23.20	18.93	25.95	3.40	20.22-26.18
X4	22.11	19.85	24.92	2.21	20.17-24.05
Y1	19.00	17.30	22.53	2.06	17.19-20.81
Y2	21.7	17.62	25.20	3.08	19.00-24.40
Y3	19.77	17.06	22.39	2.54	17.54-22.00
Y4	21.63	19.32	22.39	2.82	19.16-24.10
Z1	26.69	23.4	26.46	3.38	23.73-29.65
Z2	27.8	24.81	30.37	2.12	25.94-29.66
Z3	16.67	15.67	18.67	1.27	15.56-17.78
Z4	23.69	20.51	29.54	3.67	20.51-26.87

Shape of the investigated pores

Factor values are shown in tables and illustrated in figures. The greatest differences in the results for the aspect factor were observed for samples of series Y (1) and series of Z (1) in figure ImagePro Plus (Fig. 6), and SigmaScan figure (Fig. 7).

4. Summary

The investigations performed in the present work allow to formulate the following conclusions:

- Finer pores were observed for samples with higher concentrations of chromium and molybdenum-based powder made from CrM Astaloy than observed in the samples made on using powder Astaloy CRL. Pores of the smallest average area, smaller diameter and also smaller perimeter were observed in these samples.
- The finest pores are presented in the sample made from powder-based Astaloy CrM cooled at the rate of 6.5 °C/s and compacted at the pressure of 600 MPa. It was found that for this sample the pores have the smallest average area of pores, the smallest diameter and perimeter.
- In samples cooled at the rate of 0.3 °C/s made using powder CrL Astaloy pores are characterized by the biggest area, diameter and perimeter.

- The most similar parameters of the applied test series were obtained for samples cooled at a rate of 1.6 °C/s of the area, diameter and perimeter.
- In the analyzed samples the largest number of pores identified were characterized by the aspect factor with a value of 0.8, and thus shape similar to the round shape.
- Using the applied programs the most extreme set of the biggest and the smallest values of the area pores and the diameter for the same subjects samples were found.
- It was not possible to describe the columns in the built-in spreadsheet in SigmaScan program. In a separate document, it is necessary to save parameter names assigned to each column, as the program returns only columns with the same values. While in the program ImagePro Plus, it was found that there was no built-in spreadsheet, which requires additional software.
- The program ImagePro Plus has built more features to obtain additional information relating to the calculation of parameters individual objects. For example, the diameter in this program may be obtained as information about the maximum, minimum and average diameter calculated for every pore. While in SigmaScan program only averaged value for each pore diameter occurs.
- There are several possible scaling in the program ImagePro Plus. One of them is the scaling of images based on marker obtained during the execution images in the microscope. In the program SigmaScan there is no such possibility, so in order to calibrate the image one has to use a conversion factor calculated as the length measured in the photo section to the actual length or under the dimensions and surface area of the image.

Table 10. Assessment of statistical values for perimeter tested pores obtained using from SigmaScan program (example)

Sample symbol	Arithmetic mean, μm	Value, μm		Standard deviation, μm	Confidence interval, μm
		Min.	Max.		
X1	44.77	32.45	59.58	11.87	34.36-55.15
X2	40.88	33.51	45.95	4.85	36.63-45.13
X3	30.17	23.15	34.90	4.42	26.30-29.14
X4	27.86	26.35	30.29	1.46	26.58-29.14
Y1	25.80	26.35	30.23	3.02	23.15-28.45
Y2	27.44	23.03	33.19	4.21	23.75-31.13
Y3	23.30	22.04	27.09	2.38	21.21-25.39
Y4	24.84	21.55	29.13	2.72	22.45-27.23
Z1	37.94	22.69	42.95	3.99	34.45-41.43
Z2	39.31	33.68	46.16	4.49	35.38-43.24
Z3	21.42	35.10	24.44	2.60	19.14-23.70
Z4	30.86	18.13	27.96	2.79	28.41-33.31

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